

(I) Publication number:

0 461 758 A2

1

EUROPEAN PATENT APPLICATION

- Application number: 91304220.6
- (9) Int. CL⁵. **D06N** 7/00, C04B 26/04, C08L 9/08, C08L 25/10

- @ Date of filing: 10.05.91
- Priority: 13.06.90 US 539409
- Date of publication of application:
 18.12.91 Bulletin 91/51
- Designated Contracting States:
 BE CH DE ES FR GB IT LI LU NL SE
- (1) Applicant GENCORP INC. 175 Ghent Road Fairlawn, Ohio 44333-3300(US)
- ② Inventor: Neubert, Terry C. 5153 Sunnybrook Road Kent, Ohio 44240(US)
- (2) Representative: Stoner, Gerard Patrick et al Mewburn Eills 2 Cursitor Street London EC4A 1BQ(GB)
- © Compositions and flooring materials using them.
- A flooring felt composition comprises a fibre material such as cellulose or wood fibers, a synthetic rubber latex binder, fillers, and anticodants. The binder is a fatax copolymer made from a vinyl substituted aromatic monomer, a conjugated diene monomer, an unsaturated acid monomer, and a functional cross-linking agent offer than said acid monomer. The flooring felt is useful as a backing for lincleum, floor tile, and the like. Heat resistance of the felt is improved by excluding acrylic and methacrytic acids from the unsaturated acid monomer.

The present invention relates to a flooring felt composition useful as a backing for lineleum or floor title and containing a binder which is a latex copolymer made from a vinyl substituted aromatic monomer such as styrene, a conjugated diene monomer such as butadiene, a small amount of an unsaturated carboxylic acid such as itaconic acid, and a small amount of a functional crosslinking agent such as hydroxyothylacrylate.

BACKGROUND

Flooring felts have long been known to the art. However, a persistent problem is that they tend to be degraded by heat in drying ovens during the application and curing of a top coat to form a commercial floor covering.

Various patents exists which relate to a terpolymer emulsion, U.S. Patent No. 4,128,520; a froin-aid, U.S. Patent No. 4,172,087; various latexes as set forth in U.S. Patent Nos. 4,217,395, 4,225,383, 4,331,738, 4,436,857, 4,433,232, 4,503,184, 4,567,099, 4,782,109, and 4,857,568; an emulsion such as set forth in U.S. Patent No. 4,602,059; and a dispersion such as set forth in U.S. Patent No. 4,661,557.

Herein, we seek to provide compositions useful for new flooring materials, and preferably flooring felts of improved heat resistance.

Floaring falts of the present Invention generally contain a major amount of a generally water-insoluble filler, a styrene-butadiene type latex copolymer, various fibers, and antioxidants and have good heat resistance. More specifically, a flooring felt composition comprises approximately 100 parts by weight of one or more different types of fiber, up to about 3 parts by weight of an antioxidant, and from about 5 to about 25 parts by weight of a latex copolymer, said latex copolymer made by polymerizing a monomer moture containing (a) from about 20 to about 70 parts by weight of a vinyl substituted aromatic monomer having from 8 to 15 carbon atoms, (b) from about 30 to about 70 percent by weight of a conjugated diene having from 4 to about 12 carbon atoms, (c) up to about 5 percent by weight of an unsaturated carboxylic acid, and (d) up to about 5 percent by weight of an unsaturated carboxylic acid.

The latex copolymer which acts as a binder in a flooring felt composition is made from a vinyl substituted aromatic monomer, a conjugated dieno, an unsaturated carboxylic acid, and a functional cross-linking agent other than the carboxylic acid. Considering the vinyl substituted aromatic monomer, it may contain from 8 to about 15 carbon atoms, desirably from 8 to about 12 carbon atoms, with styrene being preferred. Examples of such monomers include alpha-methylstyrene, 3-methylstyrene vinyltoluene, propyl-styrene, butylstyrene, 1-vinylnaphthalene, 2-vinylnaphthalene, and the like. The amount of the vinyl substituted aromatic monomer is generally from about 20 to about 70 percent by weight with from about 40 to about 60 percent by weight being preferred, based upon the total weight of the latex copolymer-forming monomers.

The conjugated diene monomer utilized in forming the styrene-butadiene type copolymer is a conjugated diene having from 4 to 12 carbon atoms, desirably from about 4 to 8 carbon atoms, with butadiene being preferred. Examples of specific conjugated dienes include butadiene, isoprene, 2,3-dimethyl-1,3-butadiene, 2-methyl-1,3-pentadiene, 3,4-dimethyl-1,3-hexadiene, 4,5-diethyl-1,3-octadiene, hexadiene, and the like. The amount of the conjugated diene monomer is generally from about 30 to about 70 percent by weight and proforably from about 40 to about 60 percent by weight based upon the total weight of all the monomers forming the latex copolymer binder.

The unsaturated carboxylic acid is a polycarboxylic acid such as a dicarboxylic acid. Unsaturated carboxylic acids may be utilized which have a total of from about 4 to about 10 carbon atoms, and about 2 carboxylic acids may be utilized which have a total of from about 4 to about 10 carbon atoms, and about 2 carboxylic groups, and generally one point of unsaturation. However, it is particularly preferable that monocarboxylic acids such as acrylic acid as well as methacrylic acid and various derivatives thereof not be utilized, inasmuch as they tend to yield poor results with regard to heat resistant properties of the flooring felt composition. Thus, the latex monomer forming composition is free from acrylic acid, and the like.

Examples of sulfable unsaturated acids include fumaric acid, litaconic acid, and derivatives thereof, as well as mixtures thereof, with fumaric and itaconic acid being preferred. The amount of the unsaturated acid is generally up to about 5 percent by weight and preferably from about 1 to about 3 percent by weight based upon the total weight of the latex copolymer-forming monomers.

The functional crosslinking monomers are monomers other than the unsaturated acids which serve to form crosslinks with entities such as fibers, fillers, other latex polymer particles, and the like. Examples of functional crosslinking agents or monomers include acrylamide, methacrylamide, and N-methylolacrylamide, hydroxyethylacrylate, glycidylmethacrylate, and the like, as well as mixtures thereof. The amount of the various functional crosslinking agents utilized is up to 5 percent by weight, desirably from about 1 to about

4 percent by weight, and preferably from about 2 to about 3 percent by weight based upon the total weight of the latex copolymer-forming monomers.

The above-noted monomers forming the latex copolymer which serves as a binder may be polymerized In a conventional manner as is well known to the art and to the literature. Thus, polymerization is generally conducted in water in the presence of surfactants, chain transfer agents, various free radical initiators, various chelating agents, various shortstop compounds, electrolytes, and the like. Considering the surfactants, they can be cationic, anionic, or mixtures thereof with nonlonics. Examples of specific surfactants include the various alkyl sulfates, the various alkyl sulfosuccinates, the various alkyl aryl sulfonates, the various alpha-olofin sulfonates, the various quaternary ammonium salt, the various amine salts, the various to fatty or resin acid salts, nonyl or octyl phenol reaction products of ethylene oxide, and the like. The alkyl portion of the various surfactants generally has from 8 to 18 carbon atoms. Naturally, an amount of a surfactant is utilized to obtain an aqueous emulsion of the various monomers. Gonorally, such an amount is typically from about 0.5 to about 5 or 6 parts by weight for every 100 parts by weight of the monomers. Other surfactants can be utilized such as those set forth in McCutcheon's "Detergents and Emulsifiers," such as the 1390 edition, published by McCutcheon's Division, Allured Publishing Corporation, Ridgewood. New Jersey, "Surface Active Agents," Schwartz and Perry, Vol. I, Interscience Publishers, Inc., New York, 1958; "Surface Activity," Moilliet, Collie and Black, D. Van Nostrand Company, Inc., New York, 1961; "Organic Chemistry," Fieser and Fieser, D.C. Heath and Company, Boston, 1944; and "The Merck Index," Seventh Edition, Marck & Co., Inc., Rahway, N.J., 1960, all of which are hereby fully incorporated by reference.

The various chain extenders or molecular weight regulaturs can be conventional compounds as well as those known to the art and to the literature. Accordingly, compounds such as triphenyl methane, and carbon tetrachloride can be utilized. However, mercaptans such as the alkyl and/or aralkyl mercaptans having from 8 to about 18 curbon atoms and preferably from about 12 to about 14 carbon atoms are preferably utilized. The tertiary alkyl mercaptans having from 12 to 14 carbon atoms are highly preferred. Examples of suitable mercaptans include n-octyl mercaptan, n-dodecyl mercaptan, t-octyl mercaptan, t-dodecyl mercaptan, p-tridecyl mercaptan, tetradecyl mercaptan, hexadecyl mercaptan, and the like, as well as mixtures thereof. The amount of the molecular weight modifiers is an effective amount to provide for the proper retention of the tensile strength of the copolymer, for example from about 0.1 to about 5.0 parts by weight and desirably from about 0.2 to about 1.0 parts by weight for every 100 parts by weight of the monomers.

The free-radical initiators which are utilized to polymerize the various monomers are utilized in amounts sufficient to obtain a desired molecular weight. A suitable amount is generally from about 0.15 to about 2.0 with from about 0.25 to about 1.5 parts being preferred for every 100 parts by weight of the monomers. Conventional free-radical initiators can be utilized as well as those known to the art and to the literature. Specific examples include ammonlum persulfate, potassium persulfate, or sodium persulfate, hydrogen peroxide, and the like. Other free-radical initiators can be utilized which decompose or become active at the temperature utilized during polymerization. Examples of other free-radical catalysts include cumene hydroperoxide, dibenzoyl peroxide, diacetyl peroxide deceancyl peroxide, di-butyl peroxide, diacetyl peroxide, dictumyl peroxide, isopropyl percarbonate, di-sec-butyl peroxidicarbonate, asobisdimethylvaleronitrile, 2,2'-azobisisobutyronitrile, 2,2'-azobis-2-methyl-butyronitrile, 2,2'-azobis (methylisobutyrate), and the like, and mixtures thereof. The various persultates are generally preferred in the present techniques with the sodium salt thereof being highly preferred since it imparts better color to the resulting polymer when dried.

Cholating agents can be utilized during polymerization to the up various metal impurities as well as to subjeve a uniform polymerization. The amounts of such chelating agents are generally small such as from about 0.01 to about 0.25 parts by weight for every 100 parts by weight of the monomers. Examples of suitable chelating agents include ethylene diamine tetrascetic acid, nitrilotriacetic acid, citric acid, and their ammonium, potassium, and sodium salts.

Various shortstop compounds can also be utilized. Not only do the shortstop compounds terminate the polymerization in the reactor at desired conversion levels, but also prevent further polymerization, cross-linking, etc., cluring stripping, and the like. Examples of suitable shortstop agents include hydroquinone, sodium sulfide, hydroxyl ammonium acid sulfate, hydroxyl ammonium sulfate, sodium diethyl dithiocarbamate, diethylhydroxylamine, sodium dimethyl dithiocarbamate, potassium dimethyl dithiocarbamate, dimethylammonium dimethyldithiocarbamate, hydroxylamine sulfate plus sodiumhydrosulfite, and the like. The amount of shortstop utilized is from about 0.05 to about 0.25 parts by weight for every 100 parts by weight of said monomers.

Polymerization of the various monomers is carried out at a temperature sufficient to activate the initiators and the double bonds of the monomers. However, extremely high temperatures are avoided since

١.

they cause a run-away reaction. Too low temperatures are not desired since they retard polymerization. Suitable polymerization temperatures are from about 2°C to about 90°C, desirably from about 35°C to about 80°C, and preferably from about 65°C to about 77°C. Polymerization time will naturally vary depending upon the type of monomers utilized, the type of initiator utilized, and the degree of polymerization desired. Hence, typical polymerization times can range from about 5 to about 35 hours. Polymerization is generally carried out to completion and conducted in an acidic medium when acidic monomers are utilized. Upon completion of the reaction or the desired degree of polymerization, optional bases can be added to neutralize the latex. Examples of such optional bases include NaOH, KOH, NH4OH, and the like.

The free radical polymerization can be carried out according to any conventional method including batch, incremental, or continuous. The water used during the polymerization should be free of deleterious material and hence is often distilled or ion exchanged water. The amount of water used is sufficient to enable the formation of an emulsion and to enable proper mixing of the various ingredients as well as to obtain the desired rate and degree of polymerization, heat transfer, and the like. Upon completion of polymerization, the amount of copolymer or solids content can vary from about 10 percent to about 60 percent by weight and preferably from about 50 percent to about 55 percent by weight.

Desirably, polymerization is conducted in an inert atmosphere such as nitrogen, helium, argon, and the like, and hence it is carried out in a closed reactor. The reactor can be any conventional reactor and thus have suitable ports, agitation means, heating and cooling means, and the like. In accordance with conventional practice, the reactors utilized are generally cleaned as by flushing with water between polymerization to remove traces of various initiators, shortstops, residues, surfactants, and the like.

Once the latex copolymer has been formed, it is generally added to a slurry which contains appropriate amounts of filters and fibers. The latex copolymer can also contain an optional antioxidant in generally small amounts.

In the preparation of a flooring felt composition, a slurry containing fibers is initially prepared. The 25 various fibers are generally water insoluble and can be natural or synthetic. The fibers are typically waterdispersible and it not inherently so, dispersibility can be imparted thereto by providing a small amount of hydrophilic or ionic groups or charges on the fiber as known to the art. Generally, short fibers are preferred, that is fibers having a length generally less than 25mm (1°) and preferably less than 13m (0.5"). Generally any type of fiber may be utilized known to the flooring felt composition art as well as to the literature and examples of specific fibers include polyester fiber, nylon fiber, polyethylene fiber, glass fiber, boron fiber, graphite fiber, carbon fiber, cellulose fiber as made from cotton, wood fiber as obtained from wood pulp. ceramic titer, and the like. Especially preferred in the present invention are cellulose fibers which include fibers obtained from wood such as kraft fibers. The preparation of the fiber sturry generally involves soaking the various fibers in water and then agitating the same and applying mild heat so that the agitated slurry solution is at from about 20° C-55° C (about 70 to about 130° F). The amount of the fibers is based upon 100 parts by weight of the basic recipe (dry basis) which contains the various latex copolymers, the various fibers, the various filters, as well as the optional antioxidants, and generally is from about 5 or 6 parts to about 25 parts by weight and preferably from about 7 parts to about 15 parts by weight (dry basis) based upon 100 parts by weight (dry basis) basic recipe.

Generally, any type of filler can be utilized which is known to the flooring felt art as well as to the literature and such fillers generally have a small particle size. The amount of the various fillers is generally from about 30 to about 90 parts by weight, desirably from about 70 to about 90 parts by weight and preferably from about 80 to about 86 parts by weight based upon 100 parts by weight (dry basis) of the basic recipe, that is, the latex copolymer, the fibers, the fillers and the antioxidants. Examples of suitable fillers include various clays such as kaolin , calcium carbonate, magnesium carbonate, titanium dioxide, zinc oxide, magnesium oxide, silica, barium sulfate, calcium-based sulfate, diatomaceous earth, aluminum silicate, magnesium silicate, mica, barytes, periite, feldspar, and the like. Clay and talc are generally preferred. The various fillers are generally added to the fiber slurry under high mixing or agitation and blended therewith.

To the fiber-filler slurry is added the above described aqueous latex copolymer. The amount of the latex copolymer on a dry basis is from about 5 or 6 to about 25 parts by weight, and preferably from about 8 to about 15 parts by weight based upon 100 parts by weight of the total basic recipe.

Another basic component of the recipe is an optional but oftentimes desirable antioxidant. The amount of the antioxidant is generally small and typically up to about 2 or 3 parts by weight (dry basis) based upon 100 parts by weight (dry basis) of the basic recipe. Generally any typically antioxidant well known to the flooring felt art as well as to the literature can be utilized such as various diphenyl amines, and the like, with specific commercial examples including Wingstay L. Santo white Crystals, or any other non-staining, non-discoloring antioxidant.

The above-noted aqueous sturry, in addition to containing various fibers, various fillers, one or more latex copolymers, and the optional antioxidants, can also contain various additives such as bactericides and jungicides in effective amounts to achieve their purpose which is generally less than 0.1 parts by weight based upon 100 parts by weight (dry basis) of the basic flooring felt composition.

After addition of the various additives to the agueous flooring felt slurry, a destabilizing agent or a ilocculating agent is added to precipitate and agglomerate all of the components. Flooculating agents are known to the art and to the literature and specific examples include cationic polymers, alum, CaCl₂, MgSO₄, and the like. The amount of flooculating agent is an effective amount to generally precipitate typically all of the flooring felt composition and desirably an amount such that the supernatant is clear. Such amounts will vary from composition to composition, but can be readily determined and are known to those skilled in the art as well as to the literature.

After the aqueous flooring felt composition has been flocculated, it is generally applied to a fourdrinier wire having a fine sized screen thereon. Vacuum is applied to remove the existing water. Then the felt is dried in an oven. The flooring felt composition has been found to have unexpectedly improved high heat resistance.

The invention will be better understood by reference to the following examples.

FURNISH PREPARATION

- 25 A. Place the following into a Williams stock breaker and disintegrator bucket and allow to soak for five minutes, then refine for 60 minutes (198 gms. bleached loraft, 7000 mls. deionized water).
 - B. Pour the furnish into a 191 (5 gal) bucket and dilute to 18,750 mls. with delonized water. Pour into 571 (15 gal) bucket and add 18,750 of distilled water.
 - 1. Check freeness:(0.528% (urnish) 568 Ml. + 432 Ml. delonised water. Record freeness on work sheet (should be about 600 CSF).
 - C. Agitate sturry and heat to 40°C (100°F).

SLURRY PREPARATION

30 (This will make three handsheets)

- A. Dip out 2,500 mls. of the above slurry into a 3.91(1 gal) bucket. Begin agitating at 1,000 rpm with a high lift blade and add 12.0 gms. Narvon F-3 clay, 31.0 gms. Afton clay, 27.3 gms. Dicalite clay, 1.5 gms. OCF Glasfiber (grade 691-20-1/8).
- Slowly add 5.00 gm. (wet weight) of a 5.0% Kymene 557 (a polyacrylamide type polymer) solution to the sturry. Allow to mix about 60 seconds.
 - C. Mix up the latex/anti-oxidant solution and dilute with 75 mls. of delonized water. Add to egitating sturry.
 - 1. Latex/anti-oxidant solution
 - a. Add 14.0 gms. of dry weight latex plus 2.26 pts. (on latex dry weight) of ANTI-OXIDANT (0.69 gms. Tiarco T-558, 42.5 percent fine grind Wingstay L dispersion. Adjust amount for other A-O dispersions).
 - D. Wait 60 seconds. Start timer and add sufficient Betz 1250 (cationic) flocculent as a 0.25 percent solution to clear water. Record weight of flocculent used.
 - Agitate for 10 minutes, then add sufficient flocculent to clear slurry completely. Note amount, then agitate five more minutes and check treeness and make handsheets.

CANADIAN STANDARD FREENESS

Follow standard CSF freeness procedures using 75 ml of slurry and 925 ml of delonized water.

SHEET FORMATION

- A. Stretch a piece of cheesecloth over screen of 20 x 20 cm (8 x 8 inch) valley sheet forming mold.
- B. Close mold, add 1 liter of water to the mold.
 - C. Add 850 mls. of the sturry to the mold, stir to disperse the fibers. Start timer and open drain valve on mold. Record time it takes for water to disappear from the surface of the sheet. Record this as the drain time in seconds.

D. Pull vacuum on the mold for 30 seconds.

COUCHING

- 5 A. Open drain box, place three 24 x 24 cm (9.5" x 9.5") blotters over sheet.
 - B. Place a 20 x 20 cm (8" x 8") S.S. plate on blotters.
 - C. Place roller in center of plate, roll back and forth five times, starting and ending in center. Remove plate and blotters.
- D. Lift sheet from screen by cheesecloth and place on fresh blotter with cheesecloth up. Remove cheesecloth.

PRESSING

A. Place sheet on three blotters in center of press. Cover with release paper and press at approximately
 13.71 (13.5 tons) for 60 seconds.

DRYING

Dry at 100 °C (215 °F) for ten minutes. Turn sheet over after five minutes.

TESTING

Obtain calipor, sheet weight, density, ambient tensile and percent elongation, 180° C (360° F) hot tensile and percent elongation, stiffness, split strength, plasticizer (pick-up, ambient tensile and elongation, 180° C (380° F) hot tensile and elongation), and 215° C (420° F) heat age.

The above flooring felt had the following recipe.

TOTAL RECIPE (BASED ON DRY PARTS)

30	MATERIALS	DRY PARTS
	Bleached Kraft	13.20
	Narvon F-3 Clay	12.00
	Afton Clay	3i.00
35 .	Dicalite Clay	27.30
	OCF Glasfiber	1.50
	Latex .	14.00
	Anti-oxidant	0.293
40		(2.25 on latex)
	Kymene 557	0.25

The latex was prepared as follows:

TABLE I MATERIALS RECIPE

A. Polymerization:

Material	Percent		Pure <u>Parts</u>
Butadisne	99.40	43.250	42.991
Itaconic Acid	100.00	1.250	1.250
Potassium Persulfate	100.00	0.300	0.300
Sulfole 120	100.00	0.330	0.330
Styrene	99.70	52.000	51.844
Dowfax 2A1	45.00	0.267	0.120
Hydroxyethyl Acrylate	97.60	3.500	3.416
Hampene Na3	40.00	0.125	0.050
Aerosal A-196	40.00	3.750	1.500
Deionized Water	100.00		93.175
B. Reactor Post			•
Potassium Persulfate	100.00	0.100	0.100
Sodium Hydroxide	50.00	1.760	0.880
Drew L-198	100.00	0.150	0.150
Deionized Water	100.00		18.146
C. Post Degassing			
Proxel GXL	25.00	0.400	0.100
Deionized Water	100.00		1.150
Theoretical Solids =	47.93 p	ercent	

35

iO

20

25

40

. .

50

TABLE II CHARGE PROCEDURE

<u> </u>	E PROCEDURE			
A. Initial Charge				
	As Is Parts	Pure Parts		
Itaconic Acid	1.250:	1.250		
Hampene Na3	0.125	0.050		
Dowfax 2A1	0.267	0.120		
Aerosal A-196	3.750	1.500		
Potassium Persulfata	0.300	0.300		
Deionized Water		88.175		
Line Temperature out at 70°C (160°F).				
Styrene	7.500	7.478		
React for 45 Minutes				
Theoretical Solids = 10.84 percent				
B. First Monomer (After 45 Minutes)				
Styrene	7.417	7.395		
Sulfole 120	0.030	0.930		
Butadiene	7.208	7.165		
Theoretical Solids = 22.3	5 percent			
C. Second Monomer (After 60 Minutes at 15-17 percent TSC)				
Styrene	7.417	7.395		
Sulfole 120	0.060	0.060		
Butadiene	7.208	7.165		
Hydroxyethyl Acrylate	0.700	0.683		
Deicnized Water	70704	1.000		
Theoretical Solids = 31.3	7 percent			
D. Third Monomer (After 45 Minutes at 21-23 percent TSC)				
Styrene	7.417	7.395		
Sulfole 120	0.060	0.060		
Butadiene	7.208	7.165		
Hydroxyethyl Acrylate	0.700	0.683		
Deionized Water	*****	1.000		
Theoretical solids = 38.3	7 percent			
	Itaconic Acid Hampene Na3 Dowfax 2A1 Aerosal A-196 Potassium Persulfate Deionized Water Line Temperature out at 7 Styrene React for 45 Minutes Theoretical Solids = B. First Monomer (After Styrene Sulfole 120 Butadiene Theoretical Solids = 23.3 C. Second Monomer (After 60 Minutes at Styrene Sulfole 120 Butadiene Hydroxyethyl Acrylate Deionized Water Theoretical Solids = 31.3 D. Third Monomer (After 45 Minutes at Styrene Sulfole 120 Butadiene Hydroxyethyl Acrylate Deionized Water Theoretical Solids = 31.3	As Is Parts 1.250		

	E. Fourth Monomer (After 45 Minutes at)	26-28 percent TS	C)	
	Styrene	7.417	7,395	
5	Sulfole 120	0.060	0.060	
	Butadiene	7.208	7.165	
	Hydroxyethyl Acrylata	. 0.700	0.683	
to	Deionized Water		1.000	
	Theoretical Solids = 43.9	Theoretical Solids = 43.96 percent		
	F. Fifth Monomer (After 45 Minutes at 1	10-32 percent TS	c)	
16	S tyrene	7.416	7.194	
	Sulfole 120	0.060	0.060	
	Butadiene	7.209	7.166	
	Hydroxyathyl Acrylate	0.700	0.683	
20	Deionized Water	*****	1.000	
	Theoretical solids = 48.5] percent		
25	G. Sixth Monomer (After 45 Minutes at)	33-35 percent TS	C)	
	Styrene	7.416	7.394	
	Sulfole 120	0.060	0.060	
	Butadiene	7.209	7.166	
30	Hydroxyethyl Acrylata	0.700	0.683	
	Deionized Water	-	1.000	
	Theoretical Solids = 52.3	3 percent	÷	
3 5	H. Post Catalyst (After 45 Hinutes at 36-38 percent TSC)			
	Potassium Persulfate	0.100	0.100	
	Deionized Water		5.007	
(0	Theoretical Solids = 51.0	5 percent		
	I. Post Addition (At 49.5-50.5 percent	TSC)		
	Sodium Hydroxide	1.760	0.880	
15	Drew L-198	0.150	0.150	
	Deionized Water		8.139	
	Theoretical Solids = 49.3	1 percent	•	

Я

J. Blowover

Deionized Water Plush ---- 5.000

Theoretical solids = 48.16 percent

K. Stripping Strip to Residual Styrene Specification

L. Post Degassing Additive

Proxel GXL 0.400 0.100

Deionized Water ---- 1.150

Theoretical Solids = 47.91 percent

Deionized water includes all water in recipe.

The above latex copolymer was utilized as a binder in the above-noted preparation of a flooring felt composition.

EXAMPLE 1

10

13

A hydroxy ethyl acrylate latex was made having the recipe set forth above and prepared in accordance with the above noted charge procedure. A flooring felt composition was then made in the exact manner as set forth nereinabove with regard to the turnish preparation, sturry preparation, etc. This flooring felt composition yielded a heat resistant value of 200 seconds.

EXAMPLE 2

In an exect identical manner as set forth in Example 1, the flooring felt composition was made except that 1/2 part by weight of the styrene monomer was removed and 1/2 part by weight of an acrylic acid was substituted therefore. This composition yielded a heat resistant value of 104 seconds.

#8 EXAMPLE 3

In an exact identical manner as set forth in Example 1, the flooring felt composition was made except that 1.5 parts by weight of the styrene monomer was removed and 1.5 parts by weight of an acrylic acid was substituted therefore. This composition yielded a heat resistant value of 62 seconds.

As apparent from the above examples, the flooring felt composition embodying the invention yielded an unexpectedly high good heat resistant value. In contrast, when very small amounts by weight of an acrylic acid were substituted in the latex copolymer recipe, a dramatic reduction in the heat resistant values were obtained.

In the above Examples, the heat resistance value is the number of seconds which the felt sample of endures at 215°C (420°F) until an exotherm is noted i.e. the temperature rises above that level.

Claims

50

55

1. A flooring felt composition, comprising:

from about 30 to about 90 parts by weight of at least one filler, from about 5 to about 25 parts by weight of at least one fiber material,

optionally, up to about 3 parts by weight of an antioxidant, and from about 5 to about 25 parts by weight of a latex copolymer, said latex copolymer comprising residues of the following monomers:

- (a) from about 20 to about 70 percent by weight of a vinyl substituted aromatic monomer having from 8 to 15 carbon atoms,
- (b) from about 30 to about 70 percent by weight of a conjugated diene having from 4 to about 12 carbon atoms.

- (c) up to about 5 percent by weight of an unsaturated polycarboxylic acid, but being substantially free of any acrylic or methacrylic acid, and
- (d) up to about 5 percent by weight of a functional crosslinking agent other than said unsaturated carboxylic acid.
- 2. A composition according to claim 1 comprising 70 to 90 parts by weight of the filler.
- 3. A composition according to claim 1 or claim 2 in which the filter is clay or tale.
- A composition according to any one of claims 1 to 3 comprising 7 to 15 parts by weight of the fibre material.
 - 5. A composition according to any one of the preceding claims in which the fibre material is cellulosic.
- 15 6. A composition according to any one of the preceding claims comprising 8 to 15 parts by weight of the latex copolymer.
- 7. A composition according to any one of the preceding claims in which the monomers residual in the latex copolymer comprise 40 to 80% by weight of the vinyl substituted aromatic monomer and 40 to 60% by weight of the conjugated dione.
- A composition according to any one of the preceding claims in which the vinyl substituted aromatic monomer comprises styrene and the conjugated diene comprises buttediene.
- 25 9. A composition according to any one of the preceding claims in which the monomers residual in the latex copolymer comprise 1 to 3% by weight of the unsaturated polycarboxylic acid and 1 to 4% by weight of the functional cross-linking agent.
 - 10. A composition according to any one of the preceding claims in which the unsaturated polycarboxylic acid comprises itacorde acid, turnaric acid, or a mixture of these.
 - A composition according to any one of the preceding claims in which the functional cross-linking agont comprises acrylamide, methacrylamide, N-methylolacrylamide, hydroxyethylacrylate, and glycidylmethacrylate.
 - 12. A flooring felt obtainable by curing a composition according to any one of claims 1 to 11.
 - A flooring material comprising a substrate of a flooring felt according to claim 12, and a top flooring material layer on said substrate.
 - 14. A method of making a flooring felt, comprising curing a composition according to any one of claims 1 to 11.

55

35